



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of :

KASAHARA et al

Serial No. : 10/537,376

Group Art Unit : 1793

Filed : June 3, 2005

Examiner : ABU ALI, SHUANGYI

For : CALCIUM PHOSPHATE BASE PARTICULATE COMPOUND, PRODUCTION  
METHOD OF THE SAME, AND COMPOSITION CONTAINING THE COMPOUND

DECLARATION

Honorable Commissioner of Patents

and Trademarks

Washington, D.C. 20231

Sir:

Hidemitsu KASAHARA, a citizen of Japan residing at Maruo Calcium Company Limited of 1455, Nishioka, Uozumi-cho, Akashi-shi, Hyogo-ken, Japan, being duly sworn depose and says that:

1. I graduated from Department of Engineering of Fukui University in 1993. I was employed by MARUO CALCIUM COMPANY LIMITED on April 1, 1993. Since then, I have been engaged mainly in research and development on inorganic additives for resins and films, and synthesis methods of the fine particles up to today. I have numerous patent applications and patents on the subjects, and have delivered several papers on those subjects. In 2005, I received a degree of doctor from Fukui University.

2. I am one of inventors of the present invention. I have studied and am fully familiar with this specification and claims, the cited references and the Office Action dated October 29, 2010.

3. The prior art (US 6,663,948) was reproduced in which the deposition of a calcium phosphate compound (R) on support particles (M) was observed by TEM photographs.

### EXPERIMENTS

#### Preparation of support particles (M) (M1)

Support particles (M1) were prepared in the same manner as in Reference Example 1 of the prior art, and a TEM photograph of a sliced support particles (M1) having a petaloid porous structure is shown by FIG. A.

#### Preparation of composite particles (MR) (E1)

Composite particles (MR) (E1) were prepared by depositing a calcium phosphate compound (R) on the support particles (M1) in the same manner as in Example 1 of the prior art, and a TEM photograph of a sliced composite particles (MR) (E1) is shown by FIG. B.

The deposition steps from starting to completion were observed by TEM photographs.

(a): support particles (in a porous state),

(b): deposition in early stage (Densification with a calcium phosphate compound is slightly progressed.),

(c): deposition in later stage (Densification with a calcium phosphate compound is almost completed.),

(d): deposition is completed (Densification with a calcium phosphate compound is completed.).

As is apparent from the TEM photographs FIG. A, FIG. B and (a). (b). (c) and (d), the prior art process comprises the two-step reaction, i.e., the production step of the support particles and the deposition step of a calcium phosphate compound on the support particles.

TEM photographs of support particle (M1) and composite particle (MR) in Example 1 of the cited reference

Support particle (M1) having a petaloid porous structure

- $D_m = 0.8 \mu m$
- $\alpha_m = 1.2$
- $\beta_m = 0.6$
- $S_m = 165 m^2/g$
- $T_{m1} = 7.3\%$
- $T_{m2} = 3.4\%$



FIG. A

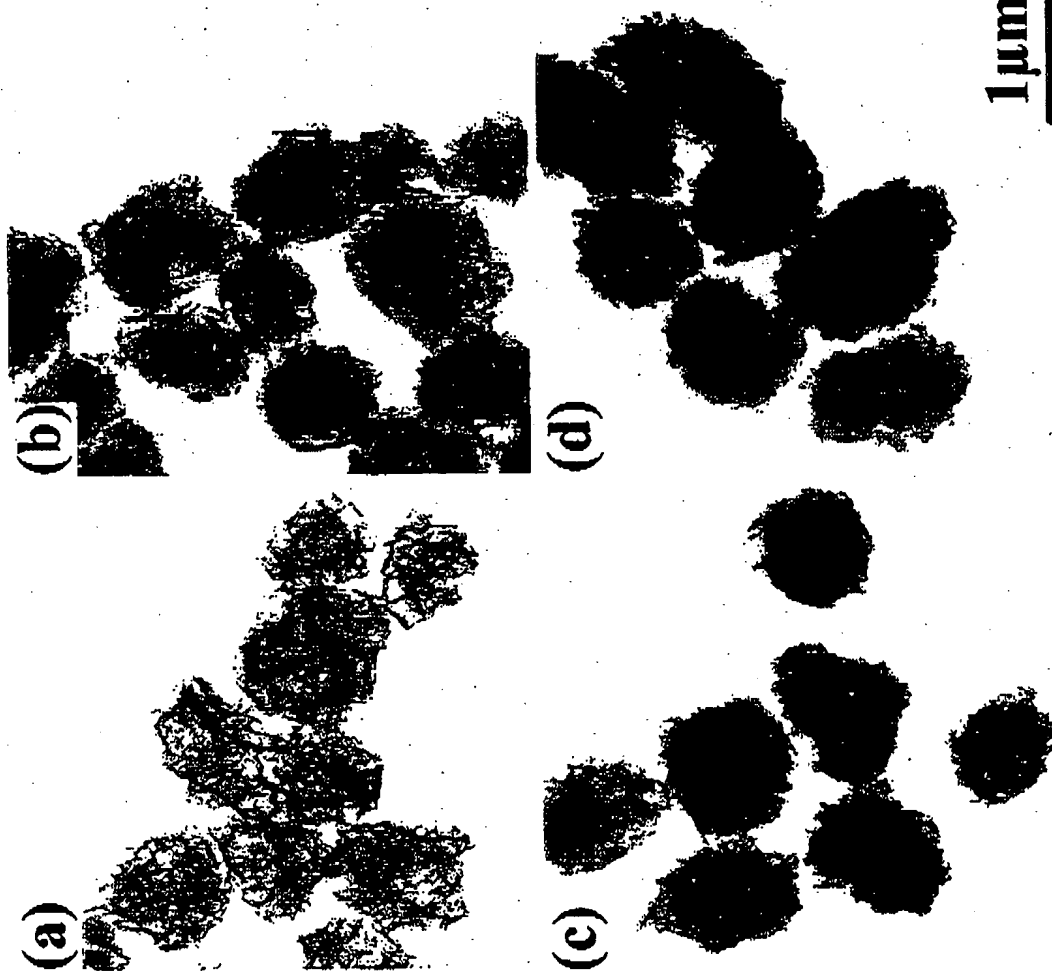
Composite particle (MR) deposited on the support particle (M1)

- $D_{mr} = 0.8 \mu m$
- $\alpha_{mr} = 1.1$
- $\beta_{mr} = 0.6$
- $s_{mr} = 75 m^2/g$
- $T_{mr1} = 3.9\%$
- $T_{mr2} = 0.8\%$



FIG. B

Time/min



TEM photographs of seeds (a) and the particles prepared by stepwise reaction at 70°C. (1 eq./60 min) ((b); 1 eq.(c); 3 eq.(d); 5 eq.)

4. All statements made herein are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated this 23rd day of February, 2011

Hidemitsu Kasahara

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2. I am one of inventors of the present invention. I have studied and am fully familiar with this specification and claims, the cited references and the Office Action dated October 29, 2010.

3. The following experiments were carried out in order to demonstrate that even when the aging is conducted under the conditions as disclosed by the prior art (US 6.663,948), calcium phosphate base particulate compound excellent in thermal stability cannot be obtained unless a heating treatment is conducted at 100 to 180°C.

#### EXPERIMENTS

##### Additional Comparative Examples 2 to 4

Calcium phosphate base particulate compounds were obtained under the conditions as disclosed by the prior art. The physical properties of the obtained compounds and production conditions are shown in Table B.



Table B

Aging conditions:

Time: 1, 12, 100 hour(s)

Temperature: 97°C

pH: 8-10

	Add. Comp. Ex. 2	Add. Comp. Ex. 3	Add. Comp. Ex. 4
(a) Sw (m <sup>2</sup> /g)	120	103	102
(b) Tg (mg/g)	132	126	123
(c) Dx50 (μm)	0.06	0.07	0.07
(d) Dx50/σx	11.5	10.4	10.0
(1) Ca conc. (wt %)	10	10	10
Kind of Ca compound	CaCl <sub>2</sub>	CaCl <sub>2</sub>	CaCl <sub>2</sub>
Complex forming substance	-	-	-
Amount (wt %)	-	-	-
(2) Phosphorus conc. (wt %)	10	10	10
Kind of phosphoric acid compound	Na <sub>2</sub> HPO <sub>4</sub>	Na <sub>2</sub> HPO <sub>4</sub>	Na <sub>2</sub> HPO <sub>4</sub>
(3) Reaction temperature (°C)	30	30	30
(4) Dropping time (min)	30	30	30
(5) Stirring blade peripheral speed (m/s)	3	3	3
(6) pH at phosphorylation	6.5-7.5	6.5-7.5	6.5-7.5
(7) Aging time (hr)	1	12	100
(9) Heating treatment temp. (°C)	97	97	97
(10) Heating treatment pH	8-10	8-10	8-10
(12) Stirring blade peripheral speed (m/s)	1	1	1
Washing filtrate conductivity (μS/cm)	150	150	150
Surface treatment agent	poly sodium acrylate	poly sodium acrylate	poly sodium acrylate
Surface treated amount (wt %)	5	5	5

As is apparent from the results of Table B. even when the aging was conducted under the conditions as disclosed by the prior art, the products excellent in thermal stability so as to meet  $1 \leq T_g \leq 100$  (mg/g) could not be obtained.

4. All statements made herein are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated this 22nd day of February, 2011

Hidemitsu Kasahara

Hidemitsu KASAHARA